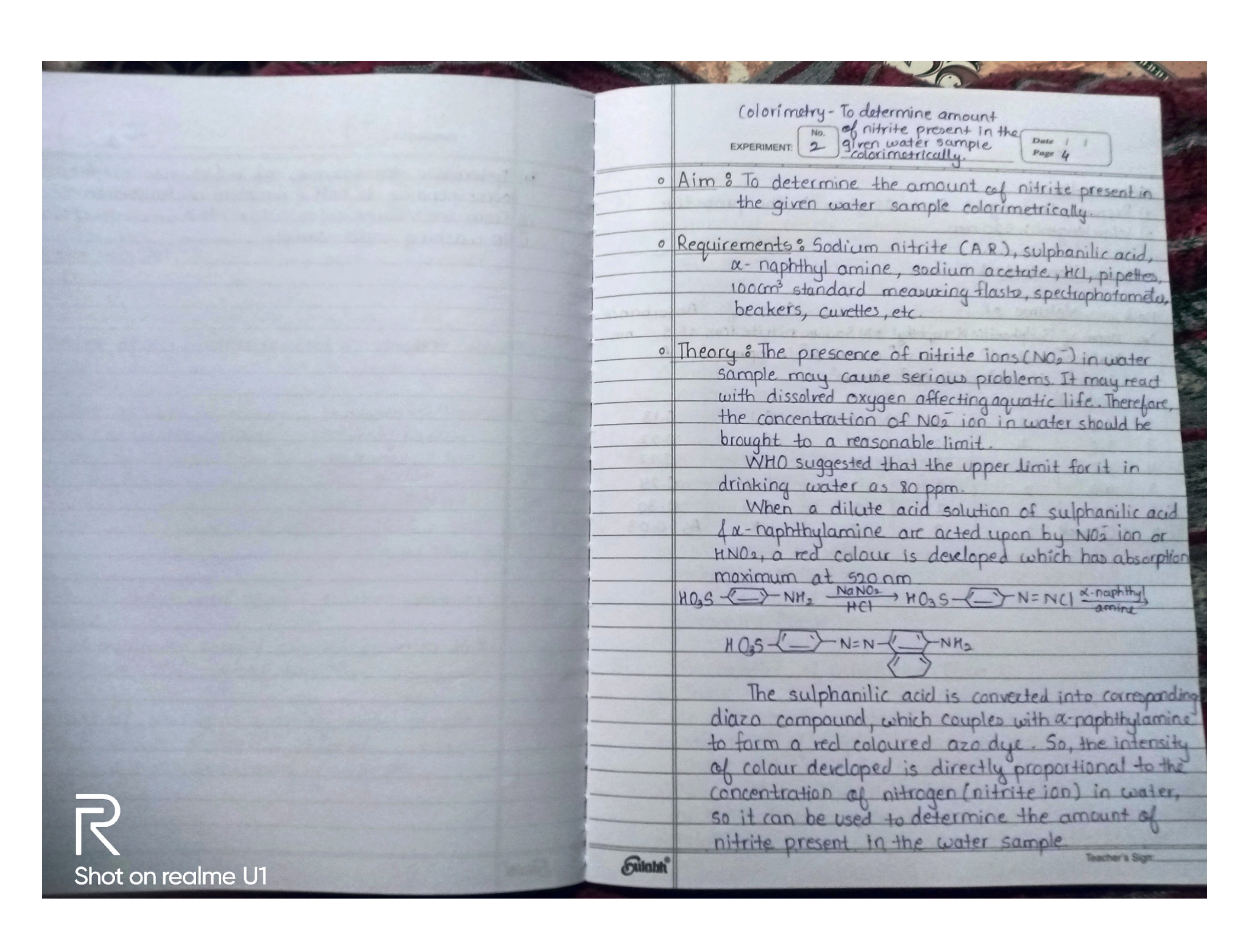


Colorimetry - To determine amount of nitrite present in the given water sample colorimetrically.

EXPERIMENT: No. 2 | Date / / | Page 4

- **Aim:** To determine the amount of nitrite present in the given water sample colorimetrically.

- **Requirements:** Sodium nitrite (A.R), sulphanilic acid, α-naphthyl amine, sodium acetate, HCl, pipettes, $100 cm^3$ standard measuring flasks, spectrophotometer, beakers, cuvettes, etc.

- **Theory:** The prescence of nitrite ions (NO_2^-) in water sample may cause serious problems. It may react with dissolved oxygen affecting aquatic life. Therefore, the concentration of NO_2^- ion in water should be brought to a reasonable limit.

  WHO suggested that the upper limit for it in drinking water as 80 ppm.

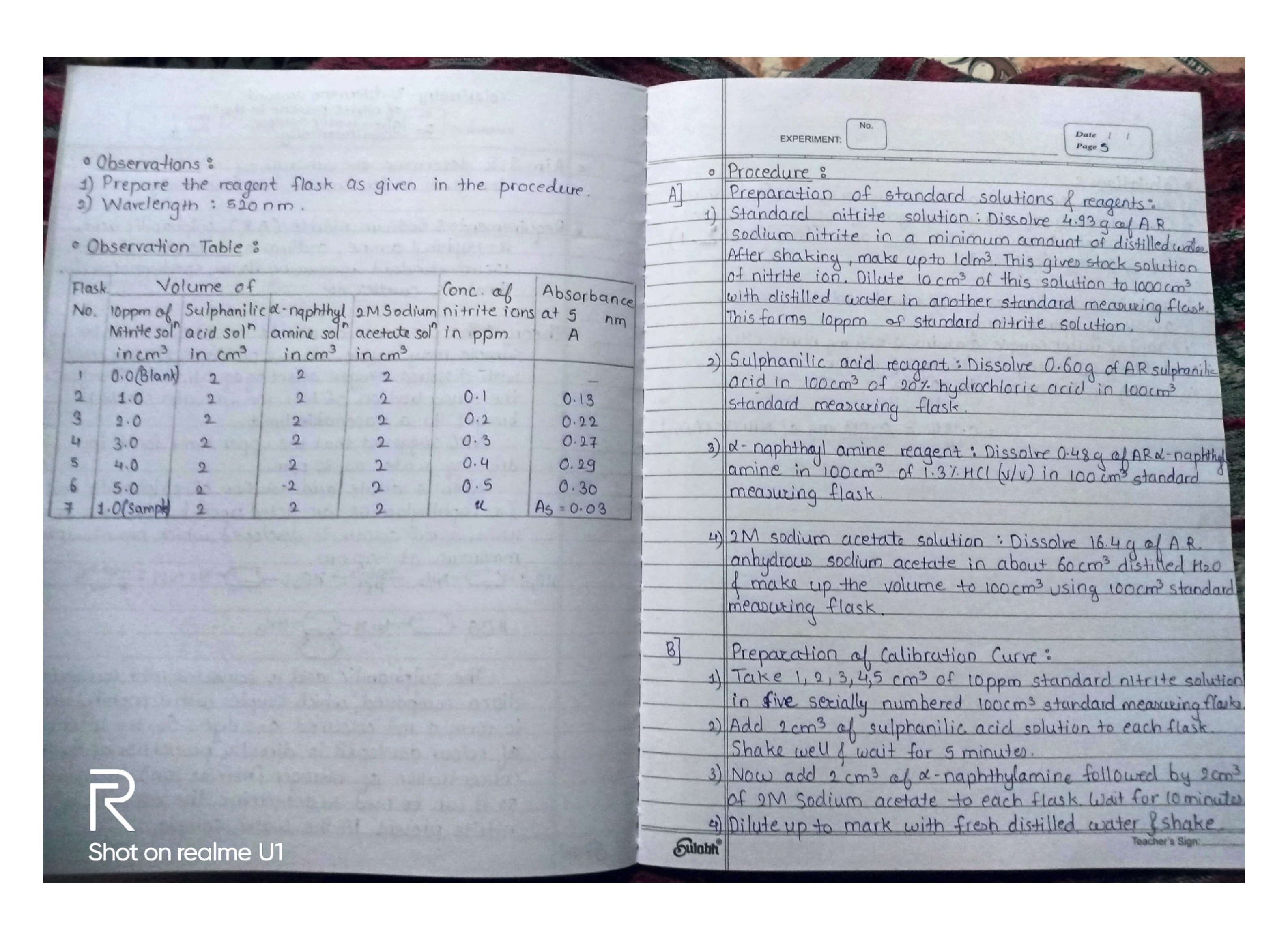
  When a dilute acid solution of sulphanilic acid & α-naphthylamine are acted upon by NO_2^- ion or HNO_2, a red colour is developed which has absorption maximum at 520 nm.

$$HO_3S-C_6H_4-NH_2 \xrightarrow[HCl]{NaNO_2} HO_3S-C_6H_4-N=NCl \xrightarrow{\alpha\text{-naphthyl amine}}$$

$$HO_3S-C_6H_4-N=N-C_{10}H_6-NH_2$$

  The sulphanilic acid is converted into corresponding diazo compound, which couples with α-naphthylamine to form a red coloured azo dye. So, the intensity of colour developed is directly proportional to the concentration of nitrogen (nitrite ion) in water, so it can be used to determine the amount of nitrite present in the water sample.

Teacher's Sign:



- **Observations :**

1) Prepare the reagent flask as given in the procedure.
2) Wavelength : 520 nm.

- **Observation Table :**

Flask No.	Volume of 10ppm of Nitrite sol^n in cm^3	Sulphanilic acid sol^n in cm^3	α-naphthyl amine sol^n in cm^3	2M Sodium acetate sol^n in cm^3	Conc. of nitrite ions in ppm	Absorbance at 5 nm A
1	0.0 (Blank)	2	2	2		–
2	1.0	2	2	2	0.1	0.13
3	2.0	2	2	2	0.2	0.22
4	3.0	2	2	2	0.3	0.27
5	4.0	2	2	2	0.4	0.29
6	5.0	2	2	2	0.5	0.30
7	1.0 (Sample)	2	2	2	x	As = 0.03

EXPERIMENT: No.

Date / /
Page 5

- **Procedure :**

A] Preparation of standard solutions & reagents:

1) Standard nitrite solution : Dissolve 4.93 g of A.R sodium nitrite in a minimum amount of distilled water. After shaking, make upto 1 dm^3. This gives stock solution of nitrite ion. Dilute 10 cm^3 of this solution to 1000 cm^3 with distilled water in another standard measuring flask. This forms 10ppm of standard nitrite solution.

2) Sulphanilic acid reagent : Dissolve 0.60 g of AR sulphanilic acid in 100 cm^3 of 20% hydrochloric acid in 100 cm^3 standard measuring flask.

3) α-naphthayl amine reagent : Dissolve 0.48 g of AR α-naphthyl amine in 100 cm^3 of 1.3% HCl (v/v) in 100 cm^3 standard measuring flask.

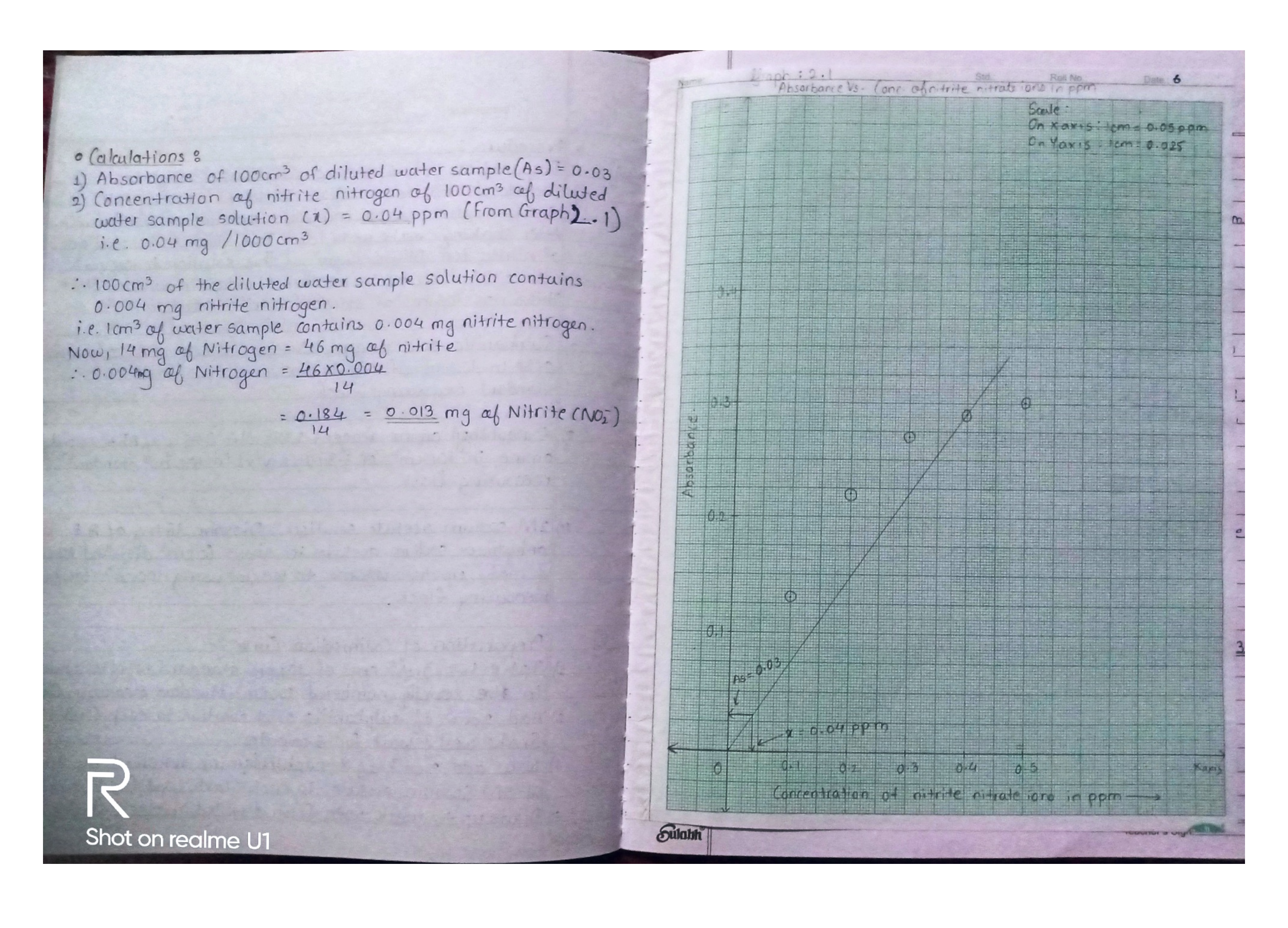
4) 2M sodium acetate solution : Dissolve 16.4 g of A.R. anhydrous sodium acetate in about 60 cm^3 distilled H_2O & make up the volume to 100 cm^3 using 100 cm^3 standard measuring flask.

B] Preparation of Calibration Curve :

1) Take 1, 2, 3, 4, 5 cm^3 of 10ppm standard nitrite solution in five serially numbered 100 cm^3 standard measuring flasks.
2) Add 2 cm^3 of sulphanilic acid solution to each flask. Shake well & wait for 5 minutes.
3) Now add 2 cm^3 of α-naphthylamine followed by 2 cm^3 of 2M Sodium acetate to each flask. Wait for 10 minutes.
4) Dilute up to mark with fresh distilled water & shake.

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Teacher's Sign:

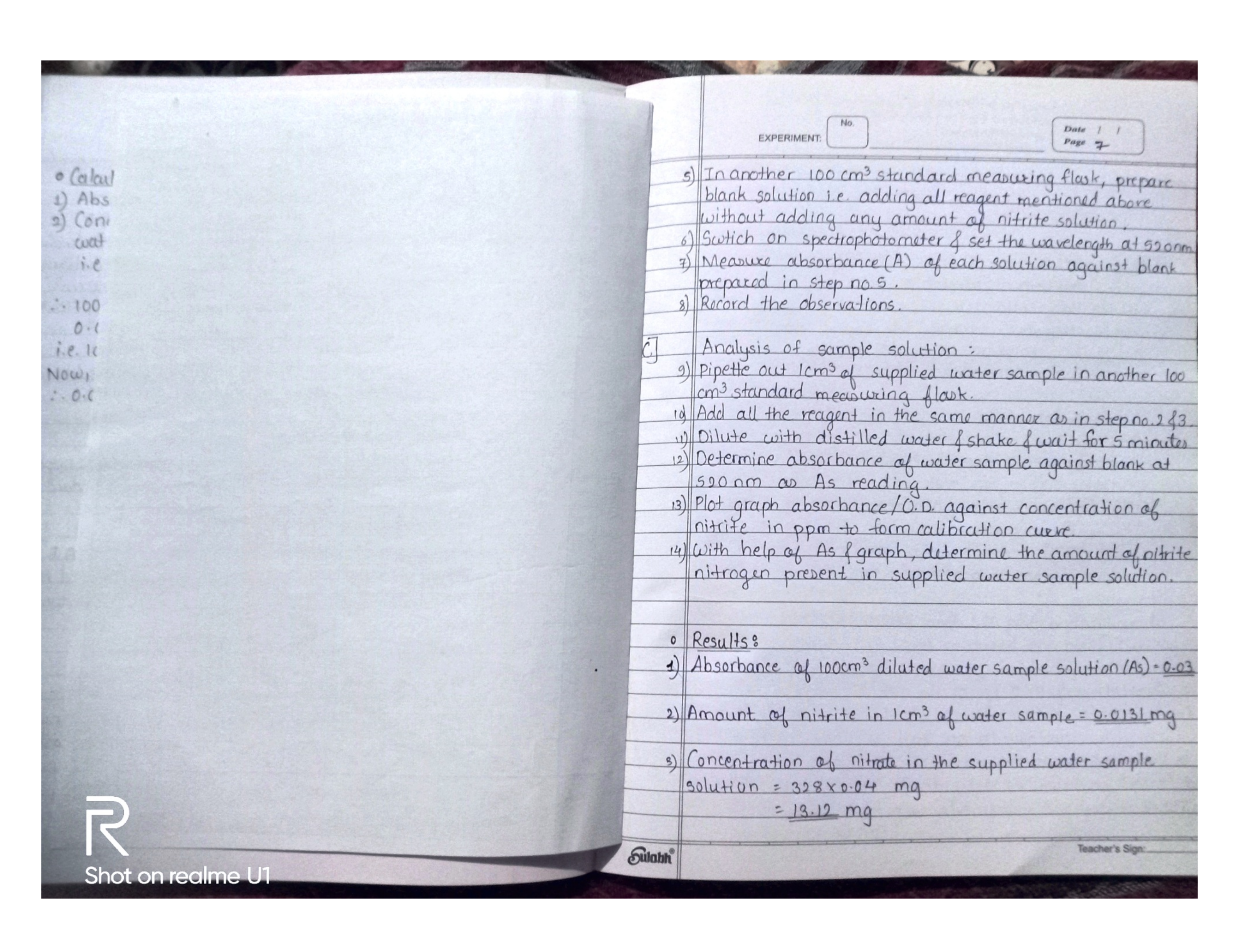


- Calculations :

1) Absorbance of $100 cm^3$ of diluted water sample $(As) = 0.03$
2) Concentration of nitrite nitrogen of $100 cm^3$ of diluted water sample solution $(x) = 0.04$ ppm (From Graph 2.1)
   i.e. 0.04 mg / $1000 cm^3$

∴ $100 cm^3$ of the diluted water sample solution contains 0.004 mg nitrite nitrogen.
i.e. $1 cm^3$ of water sample contains 0.004 mg nitrite nitrogen.
Now, 14 mg of Nitrogen = 46 mg of nitrite
∴ 0.004 mg of Nitrogen $= \frac{46 \times 0.004}{14}$

$= \frac{0.184}{14} = 0.013$ mg of Nitrite (NO_2^-)



EXPERIMENT: No. Date / / Page 7

5) In another $100\ cm^3$ standard measuring flask, prepare blank solution i.e. adding all reagent mentioned above without adding any amount of nitrite solution.
6) Swtich on spectrophotometer & set the wavelength at 520nm.
7) Measure absorbance (A) of each solution against blank prepared in step no. 5.
8) Record the observations.

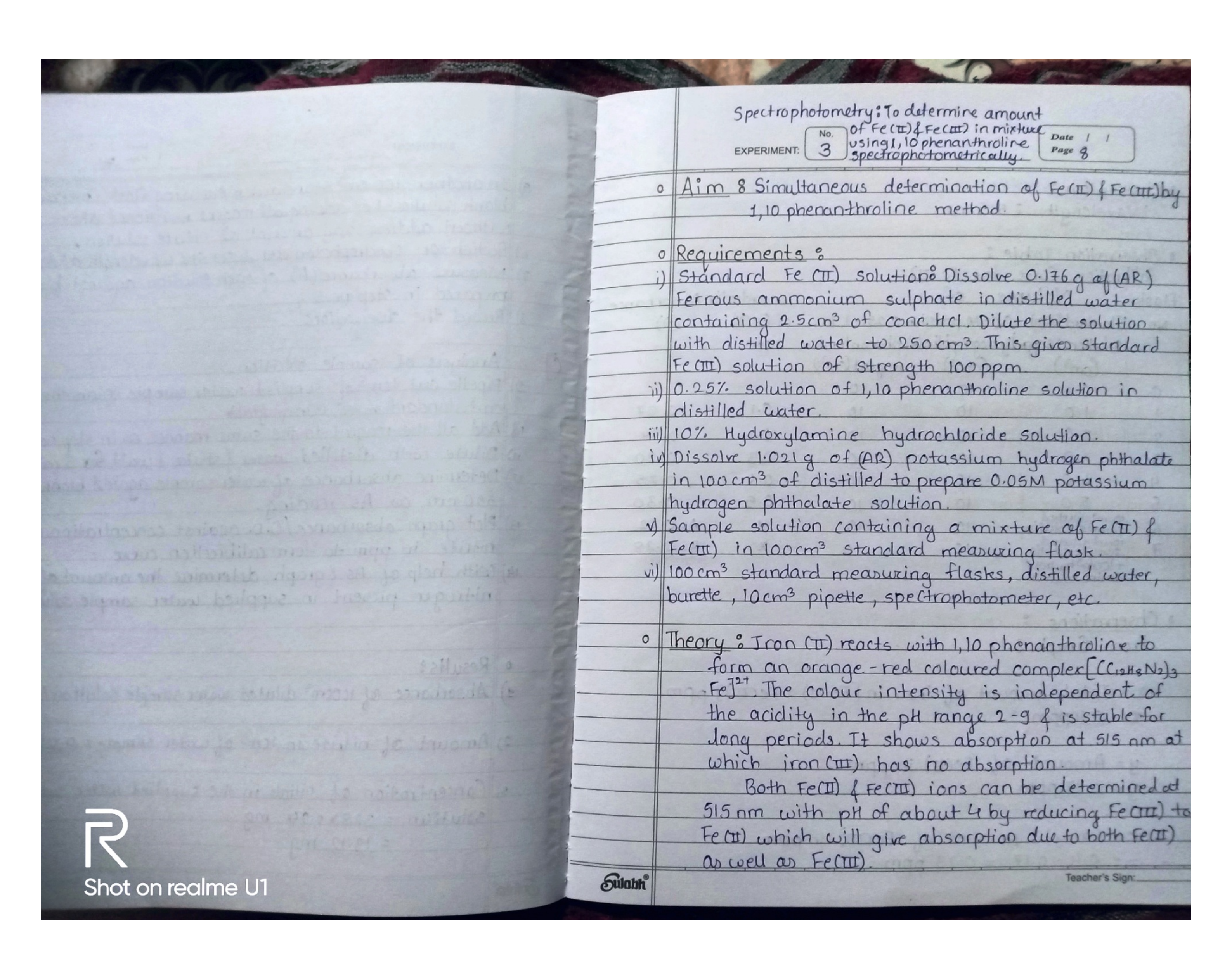
C] Analysis of sample solution :

9) Pipette out $1 cm^3$ of supplied water sample in another $100\ cm^3$ standard measuring flask.
10) Add all the reagent in the same manner as in step no. 2 & 3.
11) Dilute with distilled water & shake & wait for 5 minutes
12) Determine absorbance of water sample against blank at 520 nm as As reading.
13) Plot graph absorbance/O.D. against concentration of nitrite in ppm to form calibration curve.
14) With help of As & graph, determine the amount of nitrite nitrogen present in supplied water sample solution.

- Results :

1) Absorbance of $100cm^3$ diluted water sample solution (As) = 0.03
2) Amount of nitrite in $1cm^3$ of water sample = 0.0131 mg
3) Concentration of nitrate in the supplied water sample solution = 328×0.04 mg
= 13.12 mg

Sulabh® Teacher's Sign:


Spectrophotometry: To determine amount of Fe (II) & Fe (III) in mixture using 1,10 phenanthroline spectrophotometrically.

EXPERIMENT: No. 3

Date / /

Page 8

o **Aim**: Simultaneous determination of Fe (II) & Fe (III) by 1,10 phenanthroline method.

o **Requirements**:

i) Standard Fe (II) solution: Dissolve 0.176 g of (AR) Ferrous ammonium sulphate in distilled water containing 2.5 cm^3 of conc. HCl. Dilute the solution with distilled water to 250 cm^3. This gives standard Fe (III) solution of strength 100 ppm.

ii) 0.25% solution of 1,10 phenanthroline solution in distilled water.

iii) 10% Hydroxylamine hydrochloride solution.

iv) Dissolve 1.021 g of (AR) potassium hydrogen phthalate in 100 cm^3 of distilled to prepare 0.05M potassium hydrogen phthalate solution.

v) Sample solution containing a mixture of Fe (II) & Fe (III) in 100 cm^3 standard measuring flask.

vi) 100 cm^3 standard measuring flasks, distilled water, burette, 10 cm^3 pipette, spectrophotometer, etc.

o **Theory**: Iron (II) reacts with 1,10 phenanthroline to form an orange-red coloured complex $[(C_{12}H_8N_2)_3Fe]^{2+}$. The colour intensity is independent of the acidity in the pH range 2-9 & is stable for long periods. It shows absorption at 515 nm at which iron (III) has no absorption.

Both Fe (II) & Fe (III) ions can be determined at 515 nm with pH of about 4 by reducing Fe (III) to Fe (II) which will give absorption due to both Fe (II) as well as Fe (III).

Teacher's Sign:

Shot on realme U1

- **Observation :**

Wavelength : 515 nm

- **Observation Table :**

Calibration Curve for Fe (II):

Flask No.	Volume of			Concentration of Fe (II) ppm	Absorbance (A)
	Standard Fe(II) solution 100ppm (cm^3)	0.05M potassium hydrogen phthalate (cm^3)	0.25% 1, 10 phenanthroline reagent (cm^3)		
0	0.0	10	10	–	–
1	1.0	10	10	0.1	0.07
2	2.0	10	10	0.2	0.14
3	3.0	10	10	0.3	0.20
4	4.0	10	10	0.4	0.25
5	5.0	10	10	0.5	0.30
6	10cm^3 diluted sample solution	10	10	y	A = 0.12
7	10cm^3 diluted sample solution + 10cm^3 hydroxyl amine solution	10	10	x	B = 0.28

- **Observations :**

From Graph 3.1,

x = Total amount of iron in [Fe(II) + Fe(III)] ppm

∴ x = 0.4 ppm

y = Amount of Fe(II) in ppm

∴ y = 0.17 ppm

(x − y) = Amount of Fe(III) in ppm.

= 0.4 − 0.17 = 0.23 ppm

EXPERIMENT: No. Date / / Page 9

While another aliquote without reduction of Fe(III) to Fe(II), will give absorption due to Fe(II) only. The difference between two gives amount of Fe(III) ion.

- **Procedure :**

I. Calibration Curve for Fe(II) :

i) Take eight 100cm^3 standard measuring flasks serially numbered as 0, 1, . . . 7.

ii) Take 100ppm Fe(II) solution in a standard measuring flask No. 1 to 6 in quantity as given in Table.

iii) Add 10 cm^3 of 0.05M potassium hydrogen phthalate to each flask.

iv) Add 10 cm^3 of 0.25% 1,10 phenanthroline reagent to each flask & dilute the solution up to mark with water

v) Mix the solution well & after 10 min., determine the absorbance at 515nm against reagent blank solution in flask No. 0.

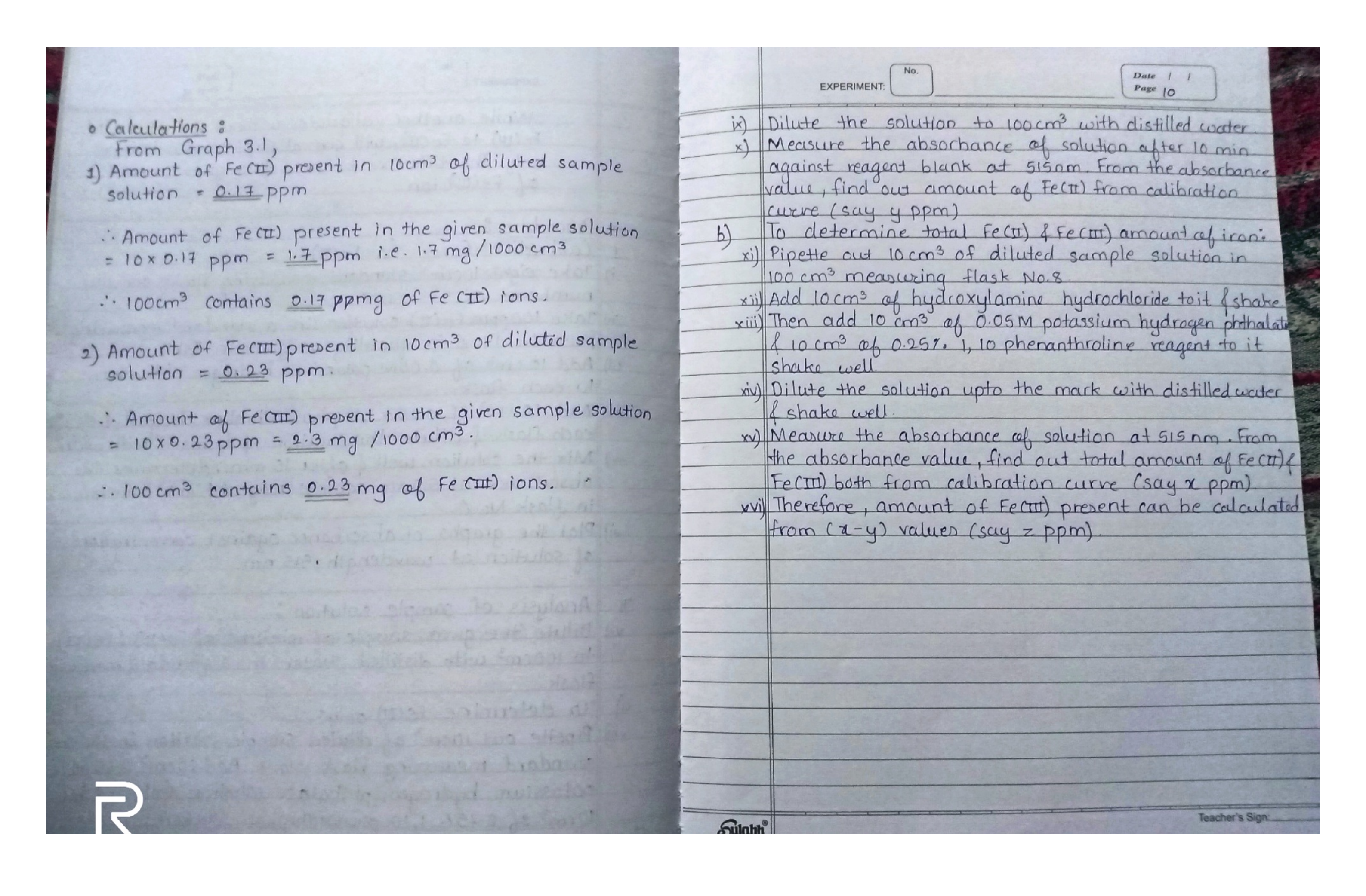
vi) Plot the graphs of absorbance against concentration of solution at wavelength 515 nm.

II. Analysis of sample solution :

vii) Dilute the given sample of mixture of Fe(II) & Fe(III) to 100cm^3 with distilled water in a standard measuring flask.

a) To determine Fe(II) only :

viii) Pipette out 10cm^3 of diluted sample solution in 100cm^3 standard measuring flask No. 7. Add 10cm^3 0.05M potassium hydrogen phthalate solution followed by 10cm^3 of 0.25% 1,10 phenanthroline solution.



• Calculations :
From Graph 3.1,

1) Amount of Fe(II) present in 10cm³ of diluted sample solution = 0.17 ppm

∴ Amount of Fe(II) present in the given sample solution
= 10 × 0.17 ppm = 1.7 ppm i.e. 1.7 mg / 1000 cm³

∴ 100cm³ contains 0.17 ppmg of Fe(II) ions.

2) Amount of Fe(III) present in 10cm³ of diluted sample solution = 0.23 ppm.

∴ Amount of Fe(III) present in the given sample solution
= 10 × 0.23 ppm = 2.3 mg / 1000 cm³.

∴ 100 cm³ contains 0.23 mg of Fe(III) ions.

EXPERIMENT: No. Date / / Page 10

ix) Dilute the solution to 100 cm³ with distilled water.

x) Measure the absorbance of solution after 10 min against reagent blank at 515nm. From the absorbance value, find out amount of Fe(II) from calibration curve (say y ppm)

b) To determine total Fe(II) & Fe(III) amount of iron:

xi) Pipette out 10 cm³ of diluted sample solution in 100 cm³ measuring flask No.8.

xii) Add 10 cm³ of hydroxylamine hydrochloride to it & shake.

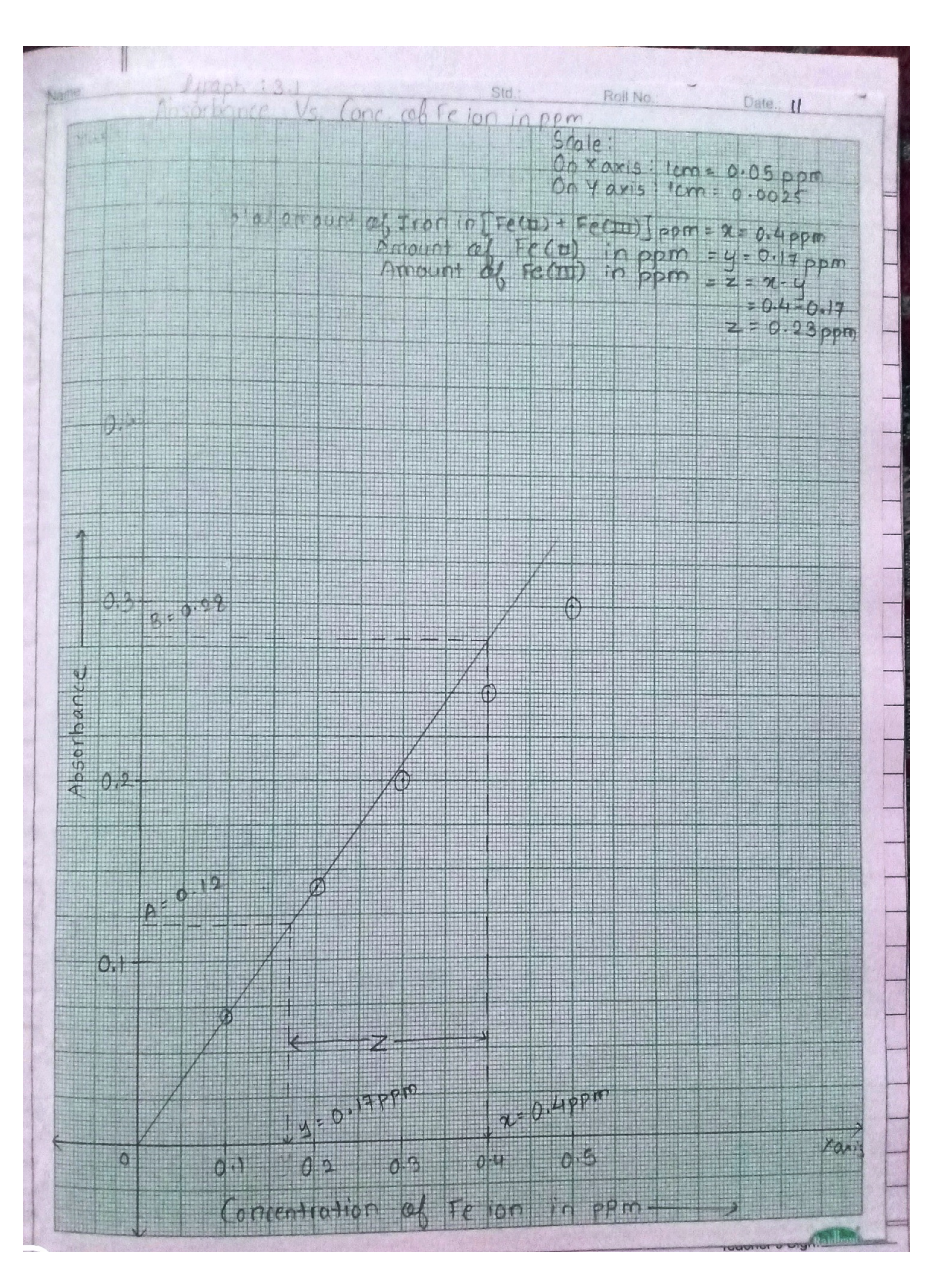
xiii) Then add 10 cm³ of 0.05M potassium hydrogen phthalate & 10 cm³ of 0.25% 1,10 phenanthroline reagent to it. Shake well.

xiv) Dilute the solution upto the mark with distilled water & shake well.

xv) Measure the absorbance of solution at 515 nm. From the absorbance value, find out total amount of Fe(II) & Fe(III) both from calibration curve (say x ppm).

xvi) Therefore, amount of Fe(III) present can be calculated from (x−y) values (say z ppm).

Teacher's Sign:


Graph : 3.1
Absorbance Vs Conc. of Fe ion in ppm.
Scale:
On X axis : 1cm = 0.05 ppm
On Y axis : 1cm = 0.0025
Total amount of Iron in [Fe(II) + Fe(III)] ppm = x = 0.4 ppm
Amount of Fe(II) in ppm = y = 0.17 ppm
Amount of Fe(III) in ppm = z = x - y
= 0.4 - 0.17
z = 0.23 ppm
B = 0.28
A = 0.12
0.3
0.2
0.1
Absorbance
Z
y = 0.17 ppm
x = 0.4 ppm
0
0.1
0.2
0.3
0.4
0.5
X axis
Concentration of Fe ion in ppm

Results :

1) Amount of Fe (II) ion in a given mixture of Fe(II) & Fe (III) = x = 0.17 mg.

2) Amount of Fe (III) ion in a given mixture of Fe(II) & Fe (III) = z = 0.23 mg.

- Observation :

I] Standardisation of 0.1N HCl Solution :

Solution in burette = 0.1 N HCl

Solution in conical flask = 10 cm^3 of diluted solution of borax + 15 cm^3 water + 1-2 drop of 1% methyl orange.

Indicator = 1% methyl orange indicator

End point = Orange to pink.

- Observation Table :

Level	Burette Reading (cm^3)			C.B.R. (cm^3)
	I	II	III	
Initial	0.0	0.0	0.0	
Final	8.1	8.0	8.0	x = 8.0 cm^3
Difference	8.1	8.0	8.0	

II] Estimation of Na_2CO_3 in Washing Soda :

Solution in burette = 0.1 N HCl

Solution in beaker = 10 cm^3 of diluted solution of sample + 25 cm^3 of distilled water.

Weight of the washing soda sample (W_1) = 0.5 g.

- Observation Table :

Obs. No.	Vol. of 0.1N HCl solution added in cm^3	Volume change ($V_2 - V_1$) in cm^3	Corrected Vol. (V_{cor}) = $V + \Delta V$ cm^3	pH of the solution	ΔpH	$\frac{\Delta pH}{\Delta V}$
1	0.0			10.40		
2	1.0	1	2	10.09	0.31	0.31
3	2.0	1	3	9.75	0.34	0.34
4	3.0	1	4	9.42	0.33	0.33
5	4.0	1	5	9.02	0.40	0.40
6	5.0	1	6	8.36	0.66	0.66
7	6.0	1	7	6.52	1.84	1.84

pH- metry : To determine percentage purity of sodium carbonate in washing soda pH metrically

EXPERIMENT: No. 1 Date / / Page 1

- Aim : To determine percentage purity of sodium carbonate in washing soda, pH metrically.

- Requirements : Washing soda powder, 0.05N borax solution, 0.1N HCl solution, 1% methyl orange indicator, buffer solution of pH 4.01 & 9.18, burette, 100 cm^3 standard measuring flasks, 100 cm^3 beakers, etc.

- Theory : Na_2CO_3 is salt of a weak acid & a strong base. As a result, in aqueous solution, it hydrolyse to form a basic solution.

The strength & amount of Na_2CO_3 can be estimated with fair accuracy using pH metric titration technique against strong acid like HCl. The reaction is given as

$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + H_2O + CO_2\uparrow$$

During the course of titration, pH is noted & graph of pH against volume of titrant added is plotted & from it, equivalence point is determined graphically.

For accurate determination of equivalence point a first derivative graph of pH/V against the volume of titrant added is plotted.

- Procedure :

A] i) Preparation of sample solution :

1) Weigh accurately 0.5 g of given sample of washing soda & transfer it to a 100 cm^3 beaker.

2) Dissolve in minimum quantity of distilled water. Transfer it to a 100 cm^3 standard measuring flask & dilute it to the mark with distilled water.

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Obs. No.	Vol. 0.1N HCl added in cm^3	$\Delta V = V_2 - V_1$	$V_{cor} = V + \Delta V$	pH	ΔpH	$\frac{\Delta pH}{\Delta V}$
8	7.0	1	8	5.84	0.68	0.68
9	8.0	1	9	5.40	0.44	0.44
10	9.0	1	10	5.04	0.36	0.36
11	10.0	1	11	4.63	0.41	0.41
12	11.0	1	12	3.50	1.13	1.13
13	12.0	1	13	1.50	2.0	2.0
14	13.0	1	14	1.16	0.54	0.54
15	14.0	1	15	0.99	0.17	0.17
16	15.0	1	16	0.85	0.14	0.14
17	15.5	0.5	16.0	0.80	0.05	0.1
18	16.0	0.5	16.5	0.75	0.05	0.1
19	16.5	0.5	17.0	0.72	0.03	0.06
20	17.0	0.5	17.5	0.67	0.05	0.1
21	17.5	0.5	18.0	0.64	0.03	0.06
22	18.0	0.5	18.5	0.62	0.02	0.04

- **Calculations :**

I] **Normality of HCl solution :**

HCl ⟶ Borax

$N_1 V_1 = N_2 V_2$

$M_1 x = 0.05 \times 25$

∴ Normality of HCl solution $= \frac{0.05 \times 25}{x}$

$A = \frac{0.05 \times 25}{8}$

$A = \underline{0.15\,N}$

A] ii) **Preparation of 0.05N borax solution :**

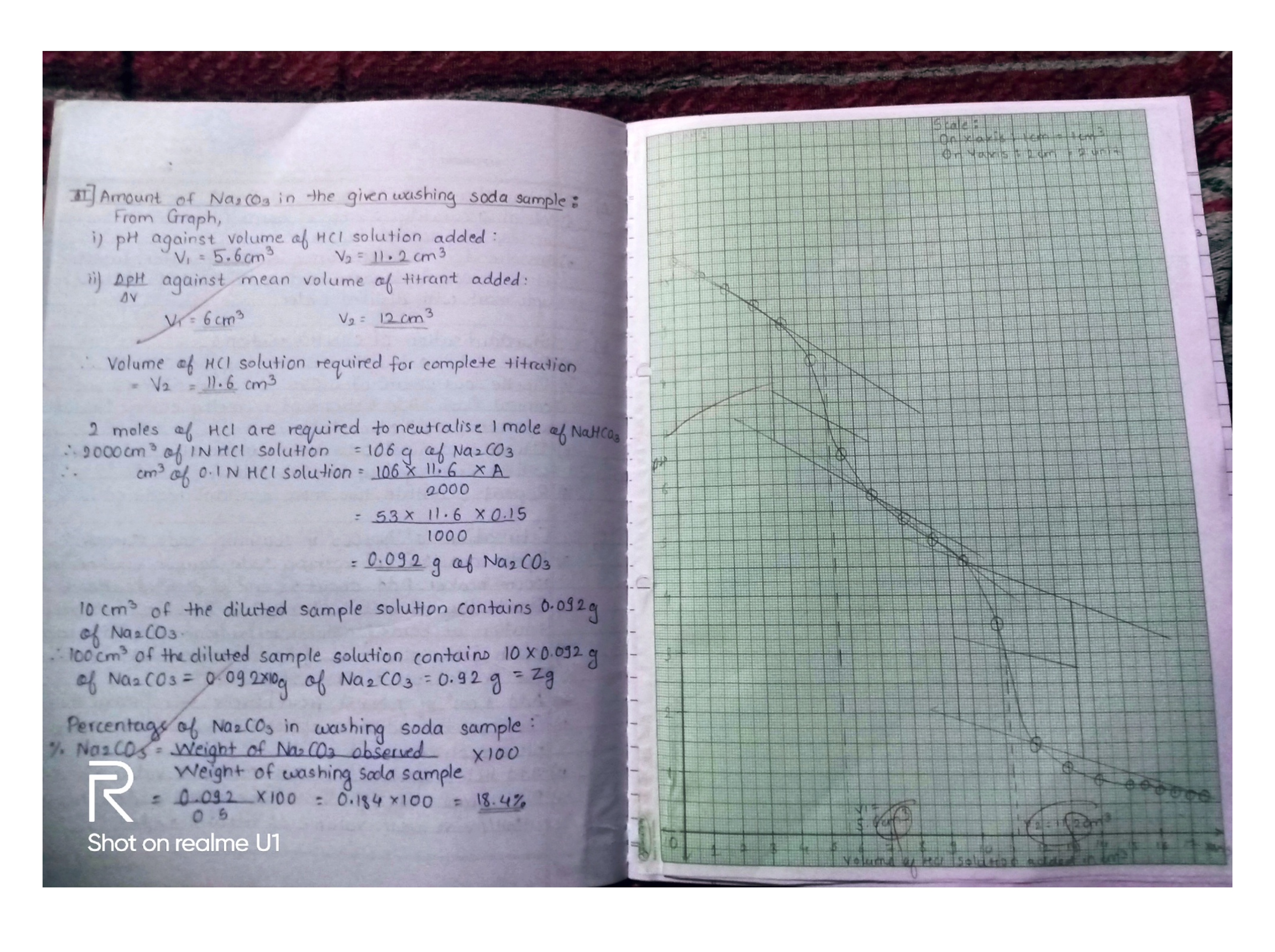
1) Weigh accurately 0.381 g of borax & transfer it to a clean dry $100 cm^3$ beaker.
2) Dissolve it in a minimum quantity of water, transfer it to a $100 cm^3$ standard measuring flask & dilute it upto mark with distilled water.

B] **Standardisation of 0.1N HCl solution :**

3) Rinse & fill a burette with the supplied 0.1N HCl.
4) Pipette out $25 cm^3$ of 0.05N borax in clean $150 cm^3$ conical flask. Add 2 drops of 1% methyl orange & about $10-15 cm^3$ distilled water to it.
5) Titrate it against 0.1N HCl from burette. End point will be from yellow to pink colour ($x\ cm^3$)
6) Repeat & obtain two more constant readings.

C] **Estimation of Na_2CO_3 in washing soda sample :**

7) Pipette out $10 cm^3$ of washing soda sample solution in $100 cm^3$ beaker. Add about $25 cm^3$ of distilled water to it.
8) Standardise pH meter at pH 9.18 & 4.01 using buffer solution of borax & potassium hydrogen phthalate respt.
9) Wash & dry electrodes & dip in sample solution & record the initial pH of the solution.
10) Add $1 cm^3$ of A N HCl from burette, stir & record the pH.
11) Continue the addition of HCl till two distinct sharp pH are observed.
12) Add HCl dropwise & till pH reaches to value about 2.
13) Plot graphs of (i) pH Vs. volume of HCl added in cm^3 (ii) $\Delta pH/\Delta V$ Vs mean volume of titrant added



II] Amount of Na_2CO_3 in the given washing soda sample :

From Graph,

i) pH against volume of HCl solution added :

$V_1 = 5.6 cm^3$ $V_2 = 11.2 cm^3$

ii) $\frac{\Delta pH}{\Delta V}$ against mean volume of titrant added :

$V_1 = 6 cm^3$ $V_2 = 12 cm^3$

∴ Volume of HCl solution required for complete titration

$= V_2 = 11.6 cm^3$

2 moles of HCl are required to neutralise 1 mole of $NaHCO_3$

∴ $2000 cm^3$ of 1N HCl solution $= 106 g$ of Na_2CO_3

∴ cm^3 of 0.1N HCl solution $= \frac{106 \times 11.6 \times A}{2000}$

$= \frac{53 \times 11.6 \times 0.15}{1000}$

$= 0.092 g$ of Na_2CO_3

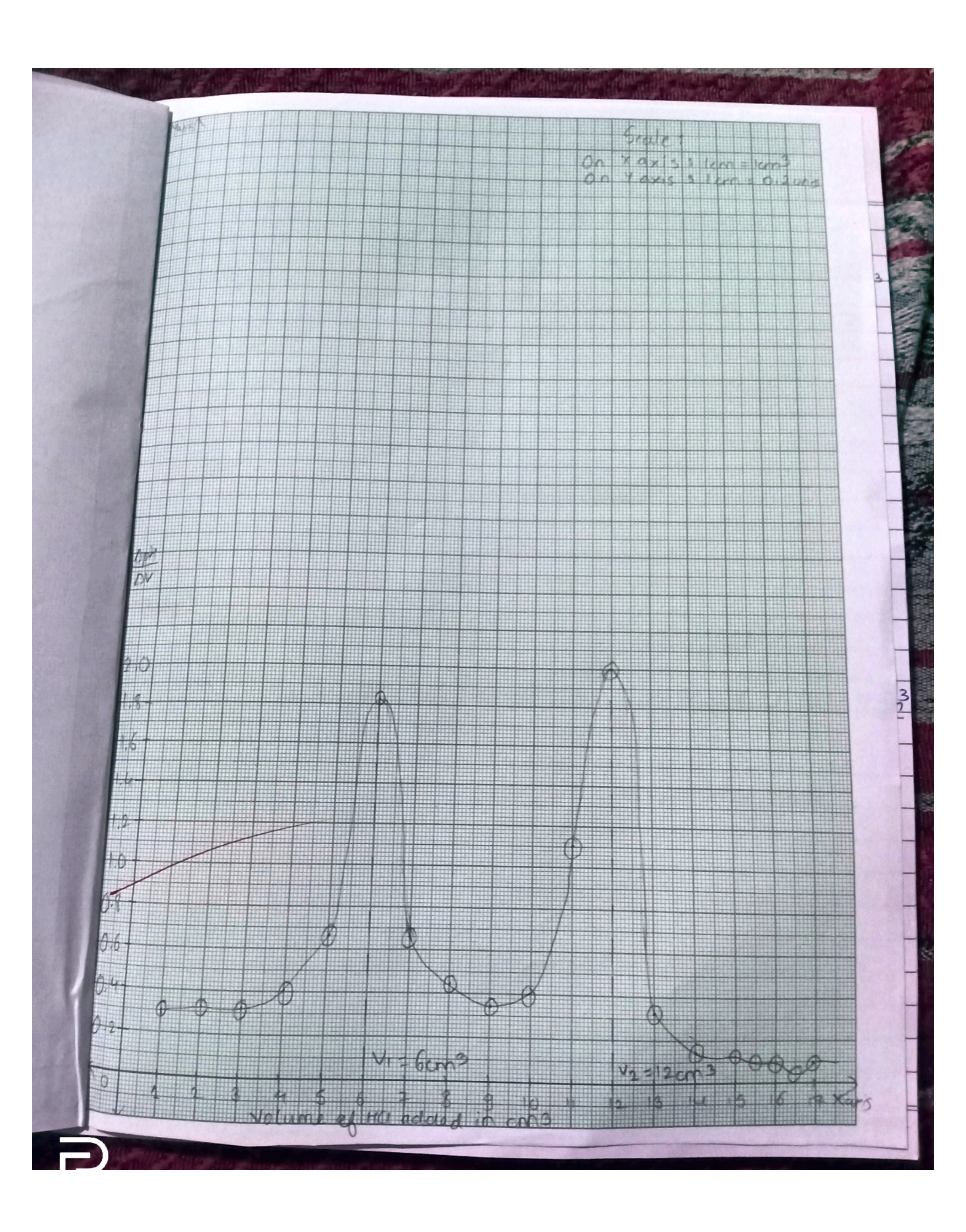
$10 cm^3$ of the diluted sample solution contains $0.092 g$ of Na_2CO_3.

∴ $100 cm^3$ of the diluted sample solution contains $10 \times 0.092 g$ of $Na_2CO_3 = 0.092 \times 10 g$ of $Na_2CO_3 = 0.92 g = Z g$

Percentage of Na_2CO_3 in washing soda sample :

$\% Na_2CO_3 = \frac{\text{Weight of } Na_2CO_3 \text{ observed}}{\text{Weight of washing soda sample}} \times 100$

$= \frac{0.092}{0.5} \times 100 = 0.184 \times 100 = 18.4\%$


Scale :
On X axis : 1cm = 1cm³
On Y axis : 1cm = 0.2 units
ΔpH/ΔV
2.0
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
X axis
V1 = 6cm³
V2 = 12cm³
Volume of HCl added in cm3

EXPERIMENT: No.

Date / /
Page 3

14) Determine the volumes of V_1 & V_2 from graph corresponding to half & complete neutralisation.

15) From the volume V_2, calculate the amount of Na_2CO_3 in washing soda sample.

o Results :

1) $10 cm^3$ of diluted sample solution required (x) = 8c of 0.15 N HCl solution

2) Percentage of Na_2CO_3 present in washing soda sample = 18.4%

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06/06/23

Teacher's Sign:

○ Observation Table :

Conc. of Cr (M)	Volume of 0.01M $K_2Cr_2O_7$	Absorbance at 440nm	Absorbance at 525 nm
0.01	10	0.416	0.055
0.0005	5	0.200	0.017
	Unknown	0.323	0.736

Conc. of Mn (M)	Volume of 0.01 M $KMnO_4$	Absorbance at 440nm	525 nm
0.00055	5.0	0.025	0.923
0.00025	2.5	0.014	0.277

○ Calculations :

I] Preparation of 0.1M $K_2Cr_2O_7$

weight = $\frac{\text{mol. wt.} \times \text{conc} \times \text{volume}}{1000}$

= $\frac{294.19 \times 0.01 \times 250}{1000}$

= 0.735 gm

II] Volume for 0.01 M $K_2Cr_2O_7$

i) $M_1V_1 = M_2V_2$

$0.01 \times V_1 = 0.001 \times 100$

$V_1 = \frac{0.001 \times 100}{0.01}$

$V_1 = 10\ cm^3$

ii) $M_1V_1 = M_2V_2$

$0.01 \times V_1 = 0.0005 \times 100$

$V_1 = \frac{0.0005 \times 100}{0.01}$

$V_1 = 5\ cm^3$

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EXPERIMENT No. 4 Determine amount of Cr (VI) & Mn (VII) in given solution by simultaneous spectrophotometrically. Date / / Page 13

○ Aim : Simultaneous determination of Chromium & Manganese in a mixture by spectrophotometrically

○ Requirements :

i) 0.005M $K_2Cr_2O_7$ standard solution :

Prepare 0.1 M $K_2Cr_2O_7$ solution by dissolving 2.942 g of $K_2Cr_2O_7$ in $100 cm^3$ standard measuring flask containing $10 cm^3$ of 10M H_2SO_4 & dilute to $100 cm^3$ distilled water. stock solution $K_2Cr_2O_7$.

ii) Dilute $5 cm^3$ stock solution to $100 cm^3$ with 1M H_2SO_4 in standard measuring flask. It gives standard 0.005M $K_2Cr_2O_7$.

ii) 0.005M $KMnO_4$ solution:

i Dissolve 0.315 g of $KMnO_4$ in $100 cm^3$ standard measuring flask containing $10 cm^3$ of 10M H_2SO_4 & dilute to $100 cm^3$ with distilled water. This gives the stock solution of $KMnO_4$.

Dilute $5 cm^3$ of stock $KMnO_4$ with 1M H_2SO_4 upto $100 cm^3$. This gives 0.005M standard $KMnO_4$.

iii) 1M H_2SO_4 solution

iv) Sample solution containing chromium (VI) & Manganese (VII) in $100 cm^3$ standard measuring flask.

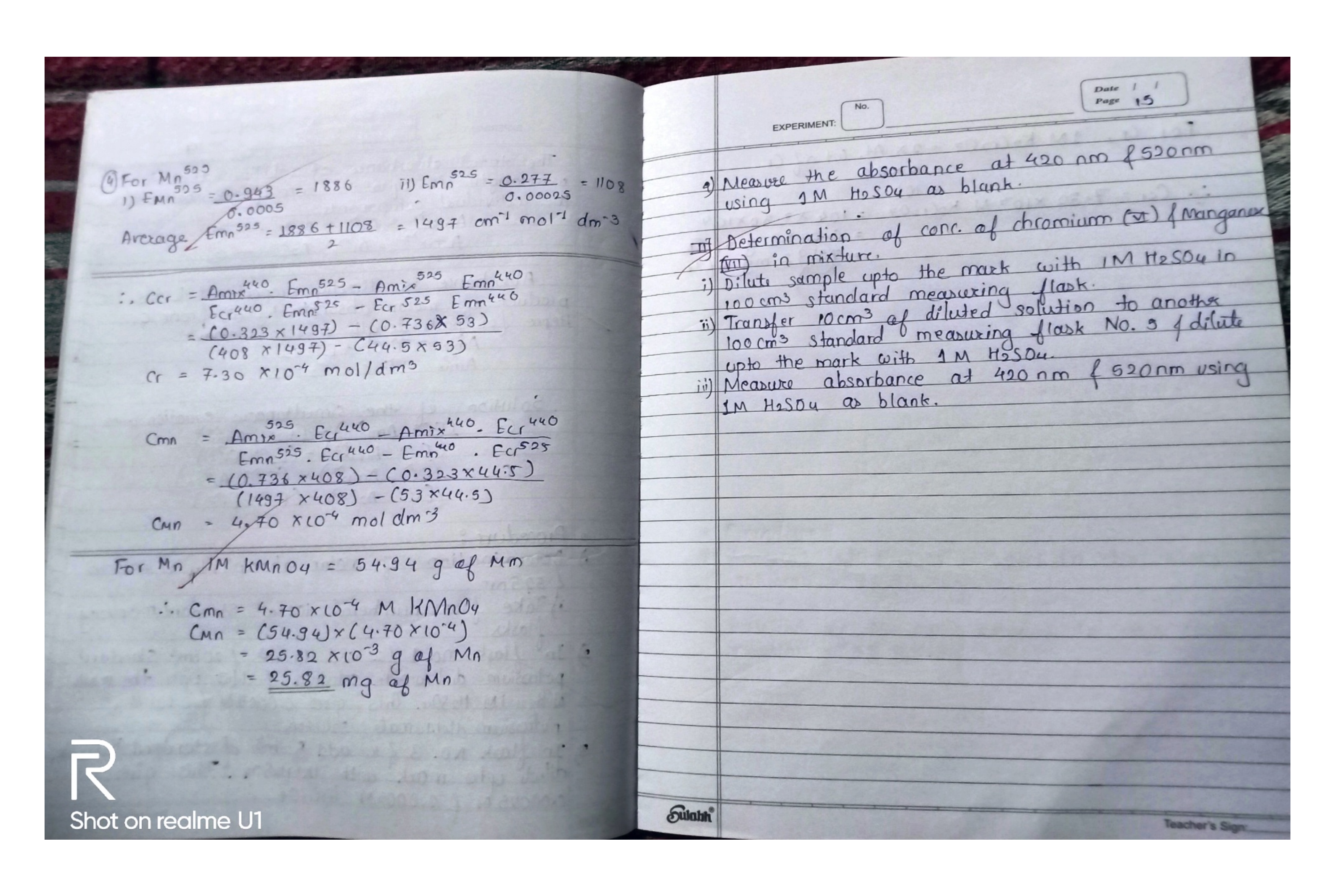
v) 6 $100 cm^3$ standard measuring flasks, burette, $5 cm^3$ & $10 cm^3$ pipette, spectrophotometer, etc.

○ Theory :

When there is no interaction between two solute in a solution it is possible to determine their conc. in mixture.

Gulab

Teacher's Sign:



(4) For Mn^{525}

i) $E_{Mn}^{525} = \frac{0.943}{0.0005} = 1886$

ii) $E_{Mn}^{525} = \frac{0.277}{0.00025} = 1108$

Average $E_{Mn}^{525} = \frac{1886 + 1108}{2} = 1497\ cm^{-1}\ mol^{-1}\ dm^{-3}$

$$\therefore C_{Cr} = \frac{A_{mix}^{440} \cdot E_{Mn}^{525} - A_{mix}^{525}\ E_{Mn}^{440}}{E_{Cr}^{440} \cdot E_{Mn}^{525} - E_{Cr}^{525}\ E_{Mn}^{440}}$$

$$= \frac{(0.323 \times 1497) - (0.736 \times 53)}{(408 \times 1497) - (44.5 \times 53)}$$

$Cr = 7.30 \times 10^{-4}\ mol/dm^3$

$$C_{Mn} = \frac{A_{mix}^{525} \cdot E_{Cr}^{440} - A_{mix}^{440} \cdot E_{Cr}^{440}}{E_{Mn}^{525} \cdot E_{Cr}^{440} - E_{Mn}^{440} \cdot E_{Cr}^{525}}$$

$$= \frac{(0.736 \times 408) - (0.323 \times 44.5)}{(1497 \times 408) - (53 \times 44.5)}$$

$C_{Mn} = 4.70 \times 10^{-4}\ mol\ dm^{-3}$

For Mn, 1M $KMnO_4$ = 54.94 g of Mn

$\therefore C_{Mn} = 4.70 \times 10^{-4}$ M $KMnO_4$

$C_{Mn} = (54.94) \times (4.70 \times 10^{-4})$

$= 25.82 \times 10^{-3}$ g of Mn

$=$ 25.82 mg of Mn

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a) Measure the absorbance at 420 nm & 520 nm using 1M H_2SO_4 as blank.

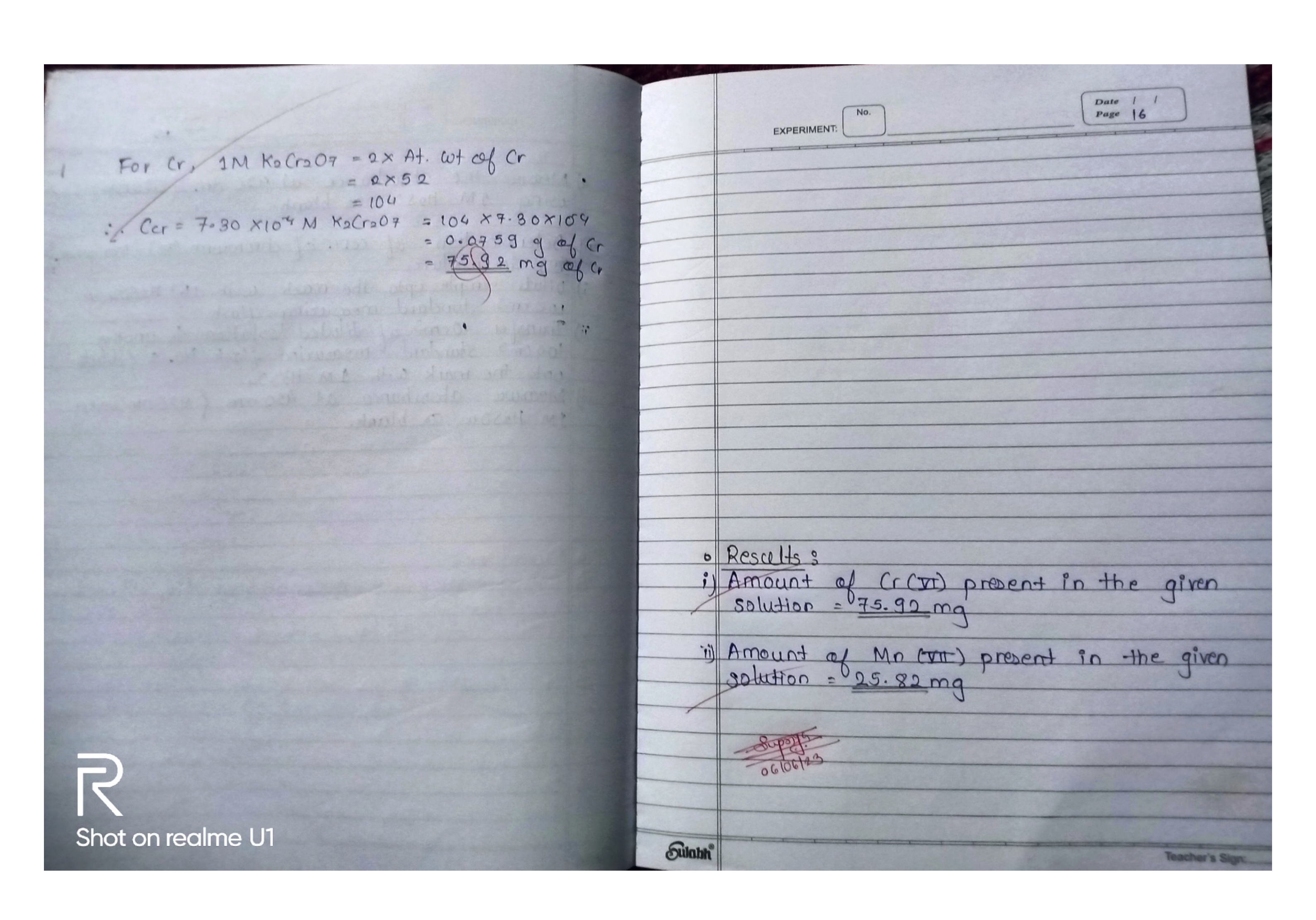
III) Determination of conc. of chromium (VI) & Manganese (VII) in mixture.

i) Dilute sample upto the mark with 1M H_2SO_4 in 100 cm^3 standard measuring flask.

ii) Transfer 10 cm^3 of diluted solution to another 100 cm^3 standard measuring flask No. 3 & dilute upto the mark with 1M H_2SO_4.

iii) Measure absorbance at 420 nm & 520 nm using 1M H_2SO_4 as blank.

Teacher's Sign:



For Cr, 1M $K_2Cr_2O_7$ = 2 × At. wt of Cr
= 2 × 52
= 104

∴ C_{Cr} = 7.30×10^{-4} M $K_2Cr_2O_7$ = $104 \times 7.30 \times 10^{-4}$
= 0.07592 g of Cr
= 75.92 mg of Cr

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- Results :

i) Amount of Cr (VI) present in the given solution = 75.92 mg

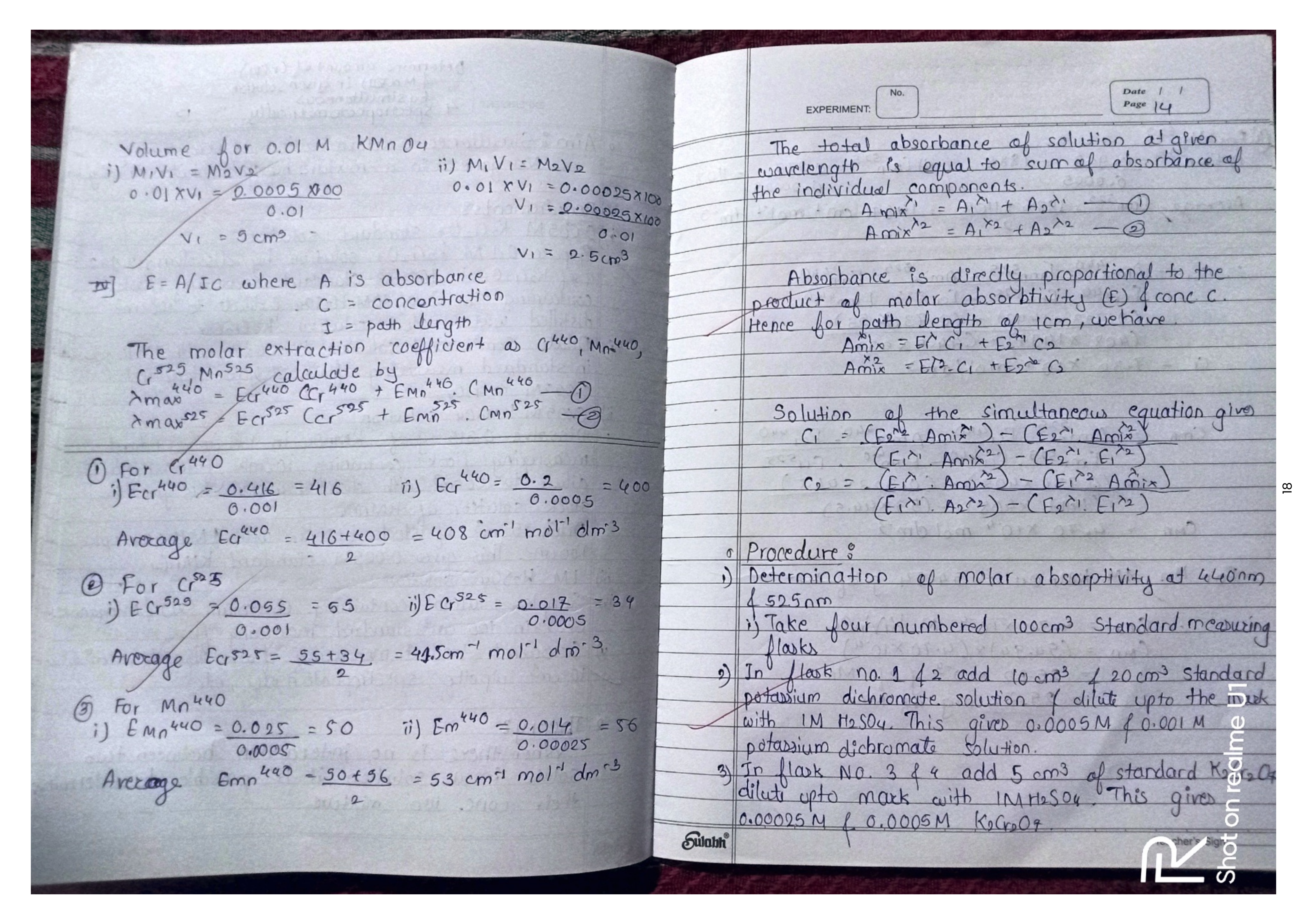
ii) Amount of Mn (VII) present in the given solution = 25.82 mg

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Teacher's Sign:

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Volume for 0.01 M $KMnO_4$

i) $M_1V_1 = M_2V_2$

$0.01 \times V_1 = \frac{0.0005 \times 100}{0.01}$

$V_1 = 5\ cm^3$

ii) $M_1V_1 = M_2V_2$

$0.01 \times V_1 = 0.00025 \times 100$

$V_1 = \frac{0.00025 \times 100}{0.01}$

$V_1 = 2.5\ cm^3$

IV] $E = A/IC$ where A is absorbance
C = concentration
I = path length

The molar extraction coefficient as Cr^{440}, Mn^{440}, Cr^{525} Mn^{525} calculate by

$\lambda max^{440} = E_{Cr}^{440}\ C_{Cr}^{440} + E_{Mn}^{440} \cdot C_{Mn}^{440}$ — (1)

$\lambda max^{525} = E_{Cr}^{525}\ C_{Cr}^{525} + E_{Mn}^{525} \cdot C_{Mn}^{525}$ — (2)

(1) For Cr^{440}

i) $E_{Cr}^{440} = \frac{0.416}{0.001} = 416$ ii) $E_{Cr}^{440} = \frac{0.2}{0.0005} = 400$

Average $E_{Cr}^{440} = \frac{416+400}{2} = 408\ cm^{-1}\ mol^{-1}\ dm^{-3}$

(2) For Cr^{525}

i) $E_{Cr}^{525} = \frac{0.055}{0.001} = 55$ ii) $E_{Cr}^{525} = \frac{0.017}{0.0005} = 34$

Average $E_{Cr}^{525} = \frac{55+34}{2} = 44.5\ cm^{-1}\ mol^{-1}\ dm^{-3}$

(3) For Mn^{440}

i) $E_{Mn}^{440} = \frac{0.025}{0.0005} = 50$ ii) $E_{Mn}^{440} = \frac{0.014}{0.00025} = 56$

Average $E_{Mn}^{440} = \frac{50+56}{2} = 53\ cm^{-1}\ mol^{-1}\ dm^{-3}$

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The total absorbance of solution at given wavelength is equal to sum of absorbance of the individual components.

$A_{mix}^{\lambda_1} = A_1^{\lambda_1} + A_2^{\lambda_1}$ — (1)

$A_{mix}^{\lambda_2} = A_1^{\lambda_2} + A_2^{\lambda_2}$ — (2)

Absorbance is directly proportional to the product of molar absorbtivity (E) & conc C. Hence for path length of 1cm, we have

$A_{mix}^{\lambda_1} = E_1^{\lambda_1} C_1 + E_2^{\lambda_1} C_2$

$A_{mix}^{\lambda_2} = E_1^{\lambda_2} C_1 + E_2^{\lambda_2} C_2$

Solution of the simultaneous equation gives

$$C_1 = \frac{(E_2^{\lambda_2} \cdot A_{mix}^{\lambda_1}) - (E_2^{\lambda_1} \cdot A_{mix}^{\lambda_2})}{(E_1^{\lambda_1} \cdot A_{mix}^{\lambda_2}) - (E_2^{\lambda_1} \cdot E_1^{\lambda_2})}$$

$$C_2 = \frac{(E_1^{\lambda_1} \cdot A_{mix}^{\lambda_2}) - (E_1^{\lambda_2} \cdot A_{mix}^{\lambda_1})}{(E_1^{\lambda_1} \cdot A_2^{\lambda_2}) - (E_2^{\lambda_1} \cdot E_1^{\lambda_2})}$$

• Procedure :

1) Determination of molar absorptivity at 440nm & 525nm

i) Take four numbered 100cm³ standard measuring flasks

2) In flask no. 1 & 2 add 10 cm³ & 20 cm³ standard potassium dichromate solution & dilute upto the mark with 1M H_2SO_4. This gives 0.0005 M & 0.001 M potassium dichromate solution.

3) In flask No. 3 & 4 add 5 cm³ of standard $K_2Cr_2O_7$ dilute upto mark with 1M H_2SO_4. This gives 0.00025 M & 0.0005 M $K_2Cr_2O_7$.

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